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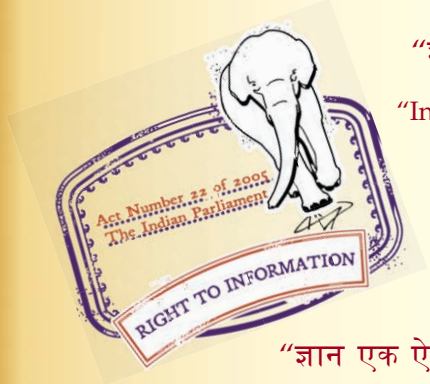

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IS : 12003 - 1987
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Indian Standard

SPECIFICATION FOR FENVALERATE, TECHNICAL

(First Reprint JUNE 1991)

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(*Continued* on page 12)

Indian Standard

SPECIFICATION FOR FENVALERATE, TECHNICAL

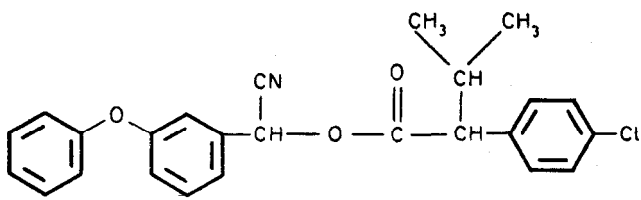
0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 28 May 1987, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Fenvalerate, technical is used for the formulation meant for the control of agricultural pests.

0.3 Fenvalerate is the accepted common name by the International Organization for Standardization (IS0) for (RS)-λ-cyano, 3-phenoxybenzyl (RS)2-(4-chlorophenyl)3-methylbutyrate. Its empirical and structural formulae and the molecular mass are given below:

Empirical Formula	*Structural Formula*	*Mvlccular Mass*
$C_{25}H_{22}ClNO_2$		419.9



0.4 In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act, 1968* and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under the Act and Rules, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed, or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of' the specifi'd value in this standard.

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for fenvalerate, technical.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of a yellowish to brown viscous liquid to semi solid mass. It shall be free from extraneous impurities or additives.

NOTE — **The** material **shall be heated on water bath before drawing samples for testing.**

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR FBNVALERATE, TECHNICAL

(*Clause* 2.2 and 5.1)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO	
			Appendix of this Standard	Cl No. of IS : 6940-1982'
(1)	(2)	(3)	(4)	(5)
i)	Fenvalerate content, percent by mass, *Min*	90.0	A	—
ii)	Acidity (as H_2SO_4), percent by mass, *Max*	1·0		11·3·2
iii)	Material insoluble in acetone, percent by mass, *Max*	0·5	—	9

***Methods of test for pesticides and their tormulations (first *revision*).**

3. PACKING AND 'MARKING

3.1 Packing — The material shall be packed according to requirements given in IS : 8190 (Part 2)-1980*.

3.2 **Marking** — The containers shall bear legibly and indelibly the following information in addition to any other information as is necessary under the *Insecticides Act, 1968* and Rules framed thereunder:

a) Name of the material;

b) Name of the manufacturer;

***Requirements for packing of pesticides: Part 2 Liquid pesticides (first *revision*).**

c) Date of manufacture;

d) Batch number;

e) Fenvalerafe content, percent (*m/m*);

f) Net mass of the contents: and

g) The minimum cautionary notice as worded in the *Insecticides Act, 1968* and Rules framed thereunder.

3.2.1 Each container may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated bv the producer., Standard marked products are also continuously checked bv BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative samples of the material shall be drawn according to IS: 10946-1984*.

NOTE — The material shall he heated on water-bath before drawing samples.

5. TESTS

5.1 Tests shall be carried out by the methods referred to in col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (see IS :1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemlcals that do not contain impurities which affect the results of analysis.

*Methods for sampling of technical grade pesticides.
†Specification for water for general laboratory use (*second revision*).

APPENDIX A

[*Table 1, Item* (i)]

DETERMINATION OF FENVALERATE CONTENT

A-O. GENERAL

A-0.1 The referee method for the estimation of fenvaierate, technical shall be the high performance liquid chromatography (HPLC) method (*see* **A-l**) or GLC method (see A-2). Apart from these method,. ultraviolet (UV) spectrophotometric method (see A-3) may be followed as a routine method. At frequent intervals, the HPLC or GLC assay shall be conducted to check the values obtained by the routine methods.

NOTE -The checking through the UV method should cover batch to batch variation, while the HPLC or GLC method should cover the variation in technical material used for making formulations.

A-l. HIGH PERFORMANCE LIQUID CHROMATOGRAPHIC METHOD

A-l.1 Principle — A HPLC unit with a UV detector is used for the assay. Using solutions containing known amounts of the standard fenvalerate sample and the internal standard (Is), the response factor (***R***) for fenvalerate in the internal standard approach is arrived at. A solution containing known mass of the fenvalerate sample (under investigation) and internal standard is injected subsequently. The percentage of fenvalerate in the sample is then computed by the standard relationship.

A-1.2 **Apparatus**

A-1.2.1 ***High Performante Liquid Chromatograph*** — equipped with a printer-plotter-cum-integrator and UV detector. The suggestive HPLC operating conditions are given below. These operating conditions are likely to change with change in the HPLC equipment employed and are allowed, provided standardization is done.

Column	Silica 25 cm x 4·6 mm (S.S)
Solvent system	a) Carbon tetrachloride — 85 percent (*v/v*) b) Chloroform — 15 percent (*v/v*) (Isocrotic)
Detector	UV (at 278 nm)
Solvent flow	1·5 ml/min
Sample concentration	Around 1 000 ppm
Sample size	10 µl

A-1.2.2 *Volumetric Flask* — 50-ml and 100-ml capacity.

A-1.2.3 *Pipettes (graduated)* — 2-ml, 5-ml and 10-ml capacity.

A-1.3 **Reagents**

A-1.3.1 *Di-n-butyl Phthalate* — AR Grade (Internal standard).

A-1.3.2 *Carbon Tetrarhloride* — Spectroscopic grade.

A-1.3.3 *Chloroform* — Spectroscopic grade.

A-1.3.4 *Standard Fenvalerate* — Of known purity *(Minimum* 92 percent).

A-1.4 Preparation of Standard Solution and Calculations of RF **and Percentage Fenvalerate**

A-1.4.1 *Preparation of Internal Standard (Is) Solution* — Weigh accurately 1·0 g of Di-n-butyl phthalate (DBP) (Is) into 100-ml volumetric flask. Dilute it up to the volume with carbon tetrachloride-chloroform (85 : 15, *v/v*) mixture. This gives 10 mg/ml of DBP solution.

A-1.4.2 ***Preparation of Standard Fenvalerate Solution for Response Factor (R F)*** - weigh accurately 0·1 g of standard fenvalerate of known purity into 100-ml volumetric flask. Add 10 ml of 'Is' solution and dilute up to the mark with solvent system. This solution gives 1 000 ppm of fenvalerate and DBP.

A-1.4.3 Introduce 10 µl of this solution to the HPLC and from the chromatogram calculate response factor as follows:

$$\text{Response factor } (Rf) = \frac{A_1}{A_2} \times \frac{m_1}{m_2}$$

where

A_1 = area of the fenvalerate,

m_1 = concentration of di-n-butyl phthalate,

A_2 = area of di-n-butyl phthalate, and

m_2 = concentration of fenvalerate.

A-1.5 Procedure

A-1.5.1 Weigh 0·5 g of fenvalerate technical sample into 100-ml volumetric flask. Make up to the volume with carbon tetrachloride-chloroform mixture. Pipette out 10 ml of the solution into 50-ml volumetric flask. Then pipette out 5 ml of 'Is' solution in the same flask. Mix well. Make up to the mark using the solvent system.

A-1.5.2 Introduce 10 µl of the above solution to the **HPLC** and, from chromatogram, calculate percentage purity of fenvalerate technical.

A-1.6 Calculations

$$\text{Fenvalerate content, percent by mass} = \frac{A_1 \times m_1 \times P \times 100}{A_2 \times m_2 \times R f}$$

where

A_1 = area of the fenvalerate peak in the sample taken for test,
m_1 = mass of 'Is' added,
P = purity of the standard,
A_2 = area of the 'Is' peak,
m_2 = mass of the sample, and
Rf = responses factor.

A-1.7 Precision — Data obtained by this method indicate a standard deviation of 0·70 for fenvalerate at 92 percent: level.

A-2. GAS CHROMATOGRAPHIC METHOD

A-2.1 Apparatus

A-2.1.1 *Gas Liquid Chromatograph* — Suitable for analysis when operated under the following suggested operating conditions with facilities for on column injection and equipped with an internal electronic integrator or equivalent. The actual set of operating parameters and instrument details used for the test and standardization shall be stated, if different from those indicated in the method:

Column	
Material	Stainless steel
Length x OD	50 cm x 0·3 mm
Stationary phase	5 percent OV-101
Solid support	Chromosorb W, HP (80 to 100 mesh)
Detector System	
Type	FID
Temperature	
Column oven	240°C
Injection Port	270°C
Detector	300°C
Carrier Gas	Nitrogen, hydrogen and air.
Carrier Gas flow	30 ml/min

A-2.1.2 *Volumetric Flask* — 50-ml and 100-ml capacity.

A-2.1.3 *Separating Funnel* — 100-ml capacity.

A-2.1.4 *Microsyringe* — 10 µl syringe with a needle of sufficient length to introduce the sample close to the top of the column packing.

A-2.2 **Reagents**

A-2.2.1 *Standard Fenvalerate* — minimum-92 percent (*m/m*).

A-2.2.2 *Di (2-ethylhexyl) Phthalate (DBP)* — AR grade.

A-2.2.3 *Chloroform* — Spectroscopic grade.

A-2.3 **Procedure**

A-2.3.1 *Preparation of Internal Standard Solution (Is) — Weigh* accurately 0·5 g DBP and dissolve in chloroform so as to make 1 litre of the solution.

A-2.3.2 *Preparation of Standard Fenvalerate Solution* — Weigh accurately 0·075 g of standard fenvalerate in a beaker and add 2·5 ml of 'Is' solution. Shake well to dissolve the fenvalerate. Transfer carefully into 50-ml volumetric flask using the 'Is' and then make up to the mark.

A-2.3.3 *Preparation of Sample Solution* — Weigh accurately about 0·075 g of fenvalerate into a 50-ml volumetric flask and make up its volume with Is.

A-2.3.4 *Estimation* — Inject 1 µl of standard fenvalerate solution (see A-2.3.2) and sample under test (see A-2.3.3). Measure the areas of fenvalerate and 'Is' peaks in each case and compute the fenvalerate content.

A-2.4 **Calculation**

$$\text{Fenvalerate content, percent by mass} = \frac{m_1 \times A_1 \times A_3 \times p}{m_2 \times A2 \times A4}$$

where

m_1 = mass in g of the standard fenvalerate,

m_2 = mass in g of the sample taken for the test,

A_1 = area of internal standard peak in standard solution,

A_2 = area of internal standard peak in sample solution,

A_3 = area of fenvalerate peak in sample solution,

A_4 = area of internal standard peak in standard solution, and

p = percentage purity of standard fenvalerate.

NOTE 1 — Peak area difference between the peaks of the standard of the sample and the internal standard should not be too large and response factor should be checked with every set of analysis.

NOTE 2 — Operational conditions and the indicated column details are suggestive and could be varied depending on GLC, provided standardization is done. In case, separation of diastereo isomers of fenvalerate is observed, peak areas of these isomers be integrated and total fenvalerate content calculated.

A-3 ULTRAVIOLET SPECTROPHOTOMETRIC METHOD

A-3.1 Prlnciple — The absorbance of a solution of the fenvalerate sample in carbon tetrachloride is measured at 278 nm against carbon ~tetrachloride blank. -The fenvalerate content of the sample is then computed making use of a calibration graph prepared earlier (using a standard fenvalerate sample).

A-3.2 **Apparatus**

A-3.2.1 *Ultraviolet Spectrophotometer*

A-3,2.2 Quartz Cells — Matched with path length equal to 1·000 cm.

A-3.2.3 *Volumetric Flasks* — 100-ml and 50-ml capacity.

A-3.2.4 *Pipette* — 5-ml and 10-ml capacity (gradutated).

A-3.3 **Reagents**

A-3.3.1 *Carbon Tetrachloride* — Spectroscopic grade.

NOTE — The absorbance of this solvent should not exceed 1.000, 0·100 and 0·005 at 263, 275 and 300 nm, respectively.

A-3.3.2 *Standard Fenvalerate* — Of known purity.

A-3.4 **Procedure** — Weigh accurately about 100 mg of standard fenvalerate sample into a 100-ml volumetric flask. Dissolve it in carbon tetrachioride and make up to volume. Pipette out 3·0, 4·0, 5·0 and 6·0 ml of this stock solution into four 50-ml volumetric flasks. Dilute them to the mark with carbon tetrachloride to obtain standard solutions of around 60, 80, 100 and 120 ppm respectively. Fill both the quartz cells with carbon tetrachloride and ensure that the cell error is insignificant (about 0.001) at *278* nm. Obtain the absorbance of the four standard solutions referred to above, at 278 nm against carbon tetrachloride blank. Use cells with lids to eliminate evaporation of carbon tetrachloride. Construct a calibration graph connecting absorbance values (corrected for cell error, if any) and concentrations of fenvalerate (in ppm) in the standard solutions. Measure the slope (S) of the calibration graph. weigh accurately about 100 mg of the fenvalerate sample under investigation into a 100-ml volumetric flask. Dissolve it in carbon tetrachloride and make up to volume. Pipette out 5'0 ml of this solution into a 50-ml volumetric flask. Dilute it to the mark with carbon tetrachloride to obtain a solution of around 100 ppm. Measure the absorbance of this solution at 278 nm against carbon tetrachloride blank. Compute the fenvalerate content of the sample making use of the calibration graph.

A-3.5 Calculation

$$\text{Fenvalerate content} = \frac{100 \times A}{M \times S}$$

where

A = absorbance of fenvalerate at 278 nm,

M = mass of the fenvalerate sample in mg taken for analysis, and

S = slope of the calibration graph.

A-3.6 **Precision** — Data obtained by this method indicate a standard deviation of 0·6 for fenvalerate at 99 percent level.

(*Continued from page 2*)

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TO
IS : 12003 - 1987 SPECIFICATION FOR FENVALERATE, TECHNICAL

(*Page 4, Table 1)* — Insert the following matter after Sl *No.* (iii) under the respective columns:

(1)	(2)	(3)	(4)	(5)
iv)	3-phenoxybenzaldehyde (MPB) content, percent [by mass. *Max*	2·0	B	—

(*Page* 11) — Insert the following appendix after A-3.6:

'APPENDIX B

[*Table 1, Item* (iv)]

DETERMINATION OF **3-PHENOXYBENZALDEHYDE** (MPB)

B-O. PRINCIPLE

B-0.1 A GIC unit with FI detector is used for this determination. Using solutions containing known amounts of the standard 3-phenoxybenzaldehyde (MPB), sample and the internalstandard (Is), the response factor, ***R,*** for MPB in the (Is) approach is arrived at. A solution containing known mass of the fenvalerate sample (under investigation), and *Is* is injected subsequently into the GIC unit. The percentage of MPB in the sample is then computed by the standard relationship.

B-l. APPARATUS

B-l.1 Gas **Liquid Chromatograph** — Suitable for analysis when operated under the following suggestive operating conditions. These conditions can be varied provided standardization is done.

Column	: 1 800 mm X **6'3** mm glass column filled with 3 percent OV-225 coated on chromosorb WHP (80-100 mesh)
Temperature of column	: 210°C
Injection port	: 160°C
Detector	: 260°C
Carrier gas	: Nitrogen at 40 ml/min

B-l.2 Volumetric Flask

B-l.3 Microsyringe

B-2. REAGENTS

B-2.1 **Metaphenoxy Beazaldebyde — AR** Grade.

B-2.2 **Cbloroform — AR** Grade.

B-2.3 Di-n-Butyl Phthalate — Internal standard (*Is*).

B-3. PREPARATION' OF STANDARDS AND CALIBRATION

B-3.1 Weigh out accurately 2·5 g of di-n-butyl phthalate into a 50-ml volumetric flask and make up to volume with chloroform. This will **give a** solution containing 50 mg/ml of the *Is*.

B-3.2 Weigh out accurately 2'5 g of standard metaphenoxy benzaldehyde into a 50-ml volumetric flask and make up to volume with chloroform. This will give a solution containing 50 mg/ml of metaphenoxy benzaldehyde. Pipette out 1'0, 2'0, 3'0, 4'0 and 5'0 ml of this metaphenoxy benzaldehyde solution into a clean dry 10-ml flask. Add 5 ml of *Is* solution in each flask. Make up the volume with chloroform in the first four cases.

B-3.3 Inject 0'2 μl of the standard solutions described under B-3.2 into the GLC unit. From the integrator print out and note down the peak areas of the metaphenoxy benzaldehyde and internal standard peaks.

B-3.4 **Procedure**

B-3.4.1 Weigh out accurately 5'0 g of the fenvalerate sample into a 10-ml volumetric flask. Measure out 5-ml of the *Is* solutions and make up to the mark with chloroform.

B-3.4.2 Introduce 0'2 μl of this sample solution the GLC unit. From the integrator print out, note down the peak areas of metaphenoxy benzaldehyde and internal standard and internal standard peaks. Compute the percentage of the metaphenoxy benzaldehyde content in the sample.

B-3.5 **Calculation**

3-phenoxybenzaldehyde content, percent by mass $= \frac{R \times A_1 \times m_1}{A \times m_2} \times 100$

where

R = response factor,

A_1 = area of the 3-phenoxybenzaldehyde peak obtained while analyzing the sample,

m_1 = mass of the internal standard added,

As = area of the internal standard peak, and

m_2 = mass of the sample taken for analysis.'

(AFCDC 6)

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AMENDMENT NO. 2 JULY 1989
TO
IS : 12003 - 1987 SPECIFICATION FOR FENVALERATE, TECHNICAL

[***Page*** *4, Table* 1 (see also Amendment No. 1)]— Delete 'SI No, (iv) and entries under the respective columns'.

(AFCDC 6)

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AMENDMENT NO. 3 APRIL 1990

TO

IS 12003 : 1987 SPECIFICATION FOR FENVALERATE, TECHNICAL

(Page 9, clause A-2.2.2) — Delete '*(DBP)*',

(Page 9, clause A-2.3.1) — Substitute the following for the existing clause:

'A-2.3.1 *Preparation of Internal Standard Solution (Is)* — Weigh accurately 0·5 g di (2-ethylhexyl) phthalate and dissolve in chloroform so as to make 1 litre of the solution.'

(*Page 9, clause* A-2.4) — Substitute 'A_4 = area of fenvalerate peak in the standard solution' *for* 'A_4 = area of internal standard peak in standard solution'.

(FADC 1)

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AMENDMENT NO. 4 SEPTEMBER 1990

TO

IS 12003 : 1987 SPECIFICATION FOR FENVALERATE, TECHNICAL

(*Page* 7, *clause* A-1 .3.4) — Substitute the following for the existing clause:

'A-1.3.4 *Reference Standard Fenvalerate* – of known purity.'

(***Page* 9, clause A-2.2.1)** — Substitute the following for the existing clause:

'A-2.2.1 Reference ***Standard Fenvale, ate*** — of known purity.'

(***Page*** 10, ***clause*** A-3.3.2) — Substitute the following for the existing clause:

'A-3.3.2 *Reference Standard Fenvalerate* — of known purity.'

(FADC 1)

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AMENDMENT NO. 5 JANUARY 2013
TO
IS 12003 : 1987 SPECIFICATION FOR
FENVALERATE, TECHNICAL

(*Page* 11, *Appendix* B (*see also Amendments No.* 1 *and* 2) — Delete.

(FAD 1)

Reprography Unit, BIS, New Delhi, India